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United States Department of Agriculture,

BUREAU OF CHEMISTRY—CIRCULAR No. 43

H. W. WILEY, Chief of Bureau.

EXTRACTS FROM THE PROCEEDINGS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1908.

[An advance circular giving the recommendations of the referees as adopted, appointments, and motions affecting the work of 1909.]

REPORTS OF COMMITTEES ON RECOMMENDATIONS OF REFEREES.

COMMITTEE A.—R. J. DAVIDSON, CHAIRMAN.

(Nitrogen, potash, phosphoric acid, soils, ash, and insecticides.)

NITROGEN.

Two recommendations (Nos. 2 and 4, Circular 38, p. 1) made by the referee in 1907, and referred to the referee for 1908, in regard to the use of copper sulphate in the Kjeldahl and Gunning methods, were again recommended for final action and were adopted. These changes in the official methods read as follows:

(1) Bulletin 107, Rev., page 6, line 4, under "(3) Determination," after the words "sulphuric acid," insert: "from 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury or in lieu of it."

(2) Bulletin 107, Rev., page 7, line 4, under "(3) Determination," after the words "sulphuric acid," insert: "from 0.1 to 0.3 gram of crystallized copper sulphate may also be added."

Recommendations (3) and (4) offered by the referee for adoption were modified as follows and proposed by the committee for further work, which latter recommendation was adopted:

(3) Bulletin 107, Rev., page 8, line 4, after the word "time," insert: "Allow the flask to stand without heat for not less than six hours, or for a shorter time, with shaking at regular intervals."

(4) Bulletin 107, Rev., page 8, under "(3) Determination," line 5, after the word "and," insert the same sentence as in recommendation (3). The sentence then reads: "Add 5 grams of thiosulphate and allow the flask to stand without heat for not less than six hours, or for a shorter time, with shaking at regular intervals; then heat the solution for five minutes," etc.

POTASH.

It is recommended—

(1) That the cobalti-nitrite method for potash be tested during the coming year (Adie and Wood, J. Chem. Soc., 77: 1076: Drushel, Amer. J. Sci., *24*: 433, or Chem. News, *97*: 124).

The Drushel modification of the cobalti-nitrite method reads as follows:

The solution of a potassium salt, containing not more than 0.2 gram of potassium oxid and free from ammonium salt, was treated with a rather large excess of sodium cobalti-nitrite solution, acidified with acetic acid, and evaporated to a pasty condition over the steam bath. It was then cooled and treated with from 50 to 100 cc of cold water, and stirred until the excess of sodium cobalti-nitrite was dissolved. It was allowed to settle, and decanted through a perforated crucible fitted with an asbestos felt. The precipitate was washed two or three times by decantation, after which it was transferred to the crucible and thoroughly washed with cold water. In the meantime a measured excess of standard potassium permanganate was diluted to ten times its volume and heated nearly to boiling. Into this the precipitate and felt were transferred and stirred, after which the crucible was also put into the solution, since particles of the precipitate stick persistently to its sides. After the oxidation had proceeded five or six minutes, manganese hydroxid separated out and the color of the solution darkened. At this point from 5 to 25 cc of sulphuric acid (1:2) were added, and the solution, after stirring, was allowed to stand a few minutes. Then a measured amount of standard oxalic acid, containing 50 cc of strong sulphuric acid per liter, was run in from a burette, taking care to add an excess. The temperature was maintained a little below the boiling point until the solution became colorless and the manganese hydroxid had completely dissolved. It was then titrated to color by permanganate in the usual manner. From the whole amount of permanganate employed the permanganate equivalent of the oxalic acid used was subtracted and the remainder multiplied by the factor calculated for the strength of permanganate used, 0.000856 being the factor for strictly tenth-normal potassium permanganate.

(2) That there be a further trial of the method involving the use of ammonium hydroxid and ammonium oxalate in the preparation of the solution in the determination of the potash in potash salts, as compared with the present method of direct precipitation of the potash without the use of the reagents mentioned.

Adopted.

The referee stated that he had not been able to take up the extensive investigations that would be necessary in attempting to define available potash, and offered the following resolution, which was passed by the association:

Resolved, That in view of the fact that practically the entire available time of the referee on potash is needed for the study of analytical methods, the investigation of the question of determining what should be designated as "available potash," provided for in a resolution adopted in 1906, be undertaken by a special referee or associate referee.

In accordance with this action the executive committee have named J. A. Bizzell, of Ithaca, N. Y., as an associate referee on potash to consider this question.

PHOSPHORIC ACID.

It is recommended—

(1) That the recommendation of 1907 be repeated, namely, that the referee on phosphoric acid shall take up for report at the next meeting of the association methods applicable under American conditions to the official examination of basic slag phosphates.

Adopted.

(2) That the referee make a further study of methods for the preparation of neutral ammonium citrate.

Adopted.

(3) That the referee investigate the amount of wash water to be employed in the treatment of the residue from the ammonium citrate digestion.

This recommendation was amended to include a study of the manner of filtering, and was so adopted.

INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the method for the separation of iron and aluminum offered as an official method be referred to the referee for 1909 for final recommendation.

Adopted.

(2) That further work be done on the sodium peroxid method for the determination of total sulphur in plants and plant products. (Bul. 107, p. 23.)

Adopted.

The method offered for the separation of iron and aluminum in ash analysis is as follows:

SEPARATION OF FERRIC AND ALUMINIC OXIDS IN INORGANIC PLANT CONSTITUENTS.

Use an aliquot of solution A, corresponding to from 0.2 to 0.5 gram of the ash, for the determination. After removing the phosphoric acid, place the filtrate from the precipitate of phosphomolybdate (consisting of the nitric acid solution of molybdic acid, ferric oxid, alumina, lime, and magnesia) in a beaker and cautiously neutralize with ammonium hydroxid, care being taken that the temperature does not rise above 40° C., and that the alkali is added only in slight excess. Allow to stand in a warm place until the precipitate completely settles. Filter the clear supernatant liquid, wash the precipitate a couple of times with hot water by decantation before transferring it to the filter, and wash four or five times on the filter with hot water. Dissolve the precipitate through the filter with weak, hot nitric acid (1:5), reprecipitate with ammonium hydroxid, filter, and wash in the same careful manner. Dry and ignite the precipitate and weigh as ferric and aluminic oxids.

Transfer an aliquot part of the original solution A, corresponding to 0.2 and 0.5 gram of ash, to an Erlenmeyer flask and evaporate with 10 cc of sulphuric acid on a hot water or steam bath until all hydrochloric acid is expelled. Dilute with distilled water to original volume and reduce the iron to the ferrous state by adding iron-free metallic zinc (about 5 decigrams at each addition) until the solution is completely decolorized and the iron is all reduced. Cool, and estimate the iron by a standard solution of potassium permanganate. Deduct the per cent of ferric oxid obtained from the per cent of ferric and aluminic oxids to obtain the per cent of alumina. Use a fiftieth-normal solution of potassium permanganate, which has been standardized by a solution of metallic iron of known composition.

SOILS.

It is recommended—

(1) That the modified J. L. Smith method for total potassium be adopted as a provisional method and be further studied. (Cir. 32, p. 4.)

This recommendation was adopted in the modified form, as presented by the committee, the referee having recommended its adoption as an optional method.

(2) That the sodium peroxid fusion for total phosphorus be continued as a provisional method and be further tested. (Bul. 105, p. 145.)

This recommendation also was adopted in the form presented by the committee, the referee having recommended the adoption of the method as official.

(3) That the magnesium nitrate method for total phosphorus be adopted as a provisional method and be further tested.

Adopted.

(4) That the Knorr method for the determination of carbonates in soils be further studied. (Wiley's Principles and Practice of Agricultural Analysis, vol. 1, ed. 1894, p. 338; ed. 1906, p. 380.)

Adopted.

The magnesium nitrate method as outlined by the referee reads as follows:

MAGNESIUM NITRATE METHOD FOR TOTAL PHOSPHURUS IN SOILS—PROVISIONAL.

Weigh into a 50 cc porcelain dish 5 grams of salt. Moisten with 5 to 7 cc of magnesium nitrate solution [Bul. 107, Rev., p. 2, sec. (g)]. Bring to dryness on the water bath. Burn off the organic matter at low redness. When cool, moisten slightly with water, add 10 cc of concentrated hydrochloric acid, and digest two hours on the water bath, keeping the dish covered with a watch glass and stirring two or three times during digestion.

Make up to 250 cc, mix well, and throw on a dry folded filter, pouring back on the filter until the solution runs through clear.

Make the determination on aliquots corresponding to 2 or 4 grams of the soil, depending upon the amount of phosphorus present. Bring to dryness, take up with hydrochloric acid and water, and filter over a pump. Filtrate and washings should not exceed 30 to 40 cc. Make alkaline with ammonium hydroxid, and dissolve the precipitate with concentrated nitric acid, using a slight excess. Add gradually, while shaking, 5 to 15 cc of molybdate solution. (Bul. 107, Rev., p. 2.) Let stand a minute or two and add 15 cc of ammonium nitrate (Bul. 107, Rev., p. 2), shaking thoroughly. Keep the solution at 40° to 50° C. for an hour, let stand over night at room temperature, filter,[a] and wash well with cold water. Put filter and precipitate back into the same flask, using as little wash water as possible. Determine phosphorus volumetrically, using standard potassium hydroxid and nitric acid.

CONVERSION TABLES.

Five conversion tables submitted by W. J. Gascoyne, of Baltimore, Md., for the consideration of the association, were referred to Committee A, which recommended that the whole question of the adoption of such tables be referred to a special committee, and after some discussion the matter was referred to the standing committee on revision of methods.

COMMITTEE B.—B. B. ROSS, CHAIRMAN.

(Dairy products, foods and feeding stuffs, sugar, tannin, and medicinal plants and drugs.)

MEDICINAL PLANTS AND DRUGS.

It is recommended—

(1) That the present provisional method for assaying opium be made official. (Bul. 107, Rev., p. 201.)

Adopted.

(2) That the methods included in the referee's report be made provisional as modified.

Adopted. [These methods were adopted provisionally in 1907 (Bul. 107, Rev., p. 258) and only the modified sections are reprinted, the changes being italicized.]

DETERMINATION OF ALKALOID.

Total extraction method.

Into a 200 cc flask weigh 10 grams of the powdered drug, add about 75 cc of ether-chloroform mixture (5 to 1 by volume), rotate, and add 5 cc of *10 per cent* ammonia water, cork, shake well and often during *two* hours.

Aliquot method.

Into a 200 cc flask weigh 15 grams of the powdered drug, add 150 cc of ether-chloroform mixture (5 to 1 by volume), cork, and shake often for several minutes. Add *5* cc of ammonia water (10 per cent), and shake frequently during *two hours.* Add 15 cc of water, or sufficient to agglomerate the drug, shake, let settle a few minutes, and then decant *100 cc* of the clear solution into a graduated cylinder.

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[a] A Hirsch funnel, with a double qualitative filter S and S No. 597, cut to fit and pressed down well around the edge after wetting and putting on pressure, is recommended.

NOTE.—Under both methods substitute "a few cubic centimeters" for the words "a small portion" referring to the ether-chloroform rinsing.

CINCHONA BARK.

Method I.

U. S. Pharmacopœia VIII, p. 102. Report total and ether-soluble alkaloids.

Method II.

Total extraction, gravimetric. *In extracting the drug let stand over night.*

(3) That the method outlined in this year's report for acetanilid mixtures be further tested, and that additional mixtures be tested by this and such other methods as may be found desirable.

Adopted. The method referred to reads as follows:

SEPARATION OF CAFFEIN, ACETANILID, AND SODIUM BICARBONATE.

Caffein.

Weigh out about 0.3 gram of headache powder on a small (5.5 cm) tared filter, wash with successive small portions of chloroform to the amount of about 30 cc, collecting the solvent in a 100 cc Erlenmeyer. Distil off chloroform by means of a small flame until only a few cubic centimeters remain. Add 10 cc of dilute sulphuric acid, (1:5) then continue the distillation till all the chloroform has gone over, disconnect from condenser, heat gently, first on wire gauze to complete solution, finally on steam or hot water bath until contents of flask have evaporated to about 3 to 4 cc. Cool, transfer by washing with water to a separatory funnel so that the final volume does not greatly exceed 20 cc. Add four times the volume, or about 80 cc, of chloroform, shake for some time vigorously, allow to stand until the chloroform clears perfectly, pass through a small dry filter into a dry 100 cc Erlenmeyer, distil off the solvent and use distillate for a second extraction, observing the same method of shaking, clearing, and filtering as above noted. Distil off chloroform to a small volume, transfer residue to a small tared beaker or crystallizing dish by means of a few cubic centimeters of chloroform. Allow to evaporate spontaneously, or, if desired, on a steam or hot water bath, to dryness, in the latter case partially covering the dish towards the end of the operation with a watch glass in order to avoid possible loss from "popping." Cool in desiccator and weigh as caffein, dry alkaloid.

Acetanilid.

First method.—The acid solution remaining in separator and containing anilin sulphate is run into a 100 cc Erlenmeyer, the filter through which the chloroform passed is washed once with a little water, allowing latter to run into the separator. Rinse the latter thoroughly, adding the aqueous rinsings to acid solution. Now run in slowly, and with constant agitation, a standard solution of potassium bromid-bromate[a] to a faint but distinct yellow coloration. The number of cubic centimeters employed, multiplied by the value of 1 cc in terms of acetanilid, will give the amount of acetanilid present.

Second method.—The aforesaid acid solution is treated with successive small portions of sodium bicarbonate until an excess of this reagent is observed in the bottom of separator. Add 50 cc of chloroform and from 15 to 20 drops of acetic anhydrid, shake for some time vigorously, allow chloroform to clear, then pass through the same filter used for the caffein into a 100 cc Erlenmeyer, and distil off most of the chloroform. Use this distillate for a second shake-out, clear, filter, and distil down to a small volume, transferring residue and subsequent chloroform washings to a tared beaker or dish precisely as in the case of caffein. Allow solvent to evaporate spontaneously or by means of a blast or fan, avoiding, however, undue heat. Dry in desiccator over quick lime to constant weight.

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[a] For this purpose the solution is prepared by adding bromin in slight excess to a concentrated aqueous solution of 50 grams of caustic potash, the liquid diluted till the separated salts redissolve, boiled to expel any excess of bromin and finally made up to 1 liter. This solution is standardized with weighed amounts of acetanilid, or it may be so adjusted by further dilution that 1 cc is exactly equivalent to 1 centigram of acetanilid. For purposes of titration 1 to 2 decigrams are heated a half hour on the steam or water bath with 10 cc of dilute sulphuric acid.

Verify final weight by means of titration with standard potassium bromid-bromate solution as in the first method. Heat residue with 10 cc of dilute sulphuric acid a half hour on steam or vapor bath, cool, add 5 cc of water and titrate as directed above.

Sodium bicarbonate.

The residue left after the first treatment with chloroform is weighed when dry and represents very nearly the amount of sodium bicarbonate present. It may be more accurately estimated by titrating with tenth-normal sulphuric acid, using congo red as indicator, or it may be ignited with dilute sulphuric acid and weighed as sodium sulphate.

Calculate results in parts per 100.

(4) That macroscopical and microscopical methods for examining drug products be studied during the coming year.

Adopted.

(5) That microchemical methods for the identification of alkaloids in drug products be further studied.

Adopted.

(6) That other microchemical methods be tested to determine the possibility of thus identifying medicinal plant principles.

Adopted.

(7) That pharmacological methods for testing the quality of drug products be investigated.

Adopted.

(8) That two associate referees on medicinal plants and drugs be appointed for the ensuing year.

Adopted.

SUGAR.

It is recommended—

(1) That the question of the influence of precipitants on the polarization of sugars be further investigated.

Adopted.

(2) That the question of methods of determining moisture or dry substance be further investigated, giving special attention to the method suggested by Doctor Horne and reported in the proceedings of 1907. (Bul. 116, pp. 22-23.)

Adopted.

(3) That the method of determining dry substance by means of a refractometer and Geerligs's table be adopted provisionally.

Adopted.

(4) That under the method for the determination of copper contained in the precipitate of cuprous oxid, pages 51–53, Bulletin 107, Revised, limit section "(6) Direct Weighing of Cuprous Oxid," page 53, by the following insertion: "This method should not be used in determining reducing sugars in commercial products, as other substances are precipitated along with the cuprous oxid. In these products the copper of the cuprous oxid should be determined direct by titration in Low's method (Bul. 107, Rev., p. 241) or as cupric oxid."

Referred to the referee for 1908–9 for investigation, with the further recommendation that the term "commercial products" be more closely defined.

Refractometer Method (Referred to in Recommendation 3).

The procedure is the same as for any work with a refractometer, and the readings are taken at 28° C. or at any other temperature. Place a few drops of the solution on the prism, adjust the border line, and read according to instructions given on page 132, Bulletin 107, Revised. Obtain the percentage of water from the table of Geerligs, which follows, and make the necessary temperature corrections indicated by the table:

Geerligs's table for dry substance in sugar-house products by the Abbe refractometer, at 28° C.[a]

Refractive index.	Per cent dry substance.	Decimals to be added for fractional readings.[b]	
1.3335	1	0.0001=0.05	0.0010=0.75
1.3349	2	0.0002=0.1	0.0011=0.8
1.3364	3	0.0003=0.2	0.0012=0.8
1.3379	4	0.0004=0.25	0.0013=0.85
1.3394	5	0.0005=0.3	0.0014=0.9
1.3409	6	0.0006=0.4	0.0015=1.0
1.3424	7	0.0007=0.5	...
1.3439	8	0.0008=0.6	...
1.3454	9	0.0009=0.7	...
1.3469	10	...	...
1.3484	11	0.0001=0.05	...
1.3500	12	0.0002=0.1	...
1.3516	13	0.0003=0.2	...
1.3530	14	0.0004=0.25	...
1.3546	15	0.0005=0.3	...
1.3562	16	0.0006=0.4	...
1.3578	17	0.0007=0.45	...
1.3594	18	0.0008=0.5	...
1.3611	19	0.0009=0.6	...
1.3627	20	0.0010=0.65	...
1.3644	21	0.0011=0.7	...
1.3661	22	0.0012=0.75	...
1.3678	23	0.0013=0.8	...
1.3695	24	0.0014=0.85	...
1.3712	25	0.0015=0.9	...
1.3729	26	0.0016=0.95	...
1.3746	27	0.0001=0.05	0.0012=0.6
1.3764	28	0.0002=0.1	0.0013=0.65
1.3782	29	0.0003=0.15	0.0014=0.7
1.3800	30	0.0004=0.2	0.0015=0.75
1.3818	31	0.0005=0.25	0.0016=0.8
1.3836	32	0.0006=0.3	0.0017=0.85
1.3854	33	0.0007=0.35	0.0018=0.9
1.3872	34	0.0008=0.45	0.0019=0.95
1.3890	35	0.0009=0.4	0.0020=1.0
1.3909	36	0.0010=0.5	0.0021=1.0
1.3928	37	0.0011=0.55	...
1.3947	38	...	...
1.3966	39	...	...
1.3984	40	...	...
1.4003	41	...	...
1.4023	42	0.0001=0.05	0.0012=0.6
1.4043	43	0.0002=0.1	0.0013=0.65
1.4063	44	0.0003=0.15	0.0014=0.7
1.4083	45	0.0004=0.2	0.9015=0.75
1.4104	46	0.0005=0.25	0.0016=0.8
1.4124	47	0.0006=0.3	0.0017=0.85
1.4145	48	0.0007=0.35	0.0018=0.9
1.4166	49	0.0008=0.4	0.0019=0.95
1.4186	50	0.0009=0.45	0.0020=1.0
1.4207	51	0.0010=0.5	0.0021=1.0
1.4228	52	0.0011=0.55	...
1.4249	53	...	...
1.4270	54	...	...
1.4292	55	0.0001=0.05	0.0013=0.55
1.4314	56	0.0002=0.1	0.0014=0.6
1.4337	57	0.0003=0.1	0.0015=0.65
1.4359	58	0.0004=0.15	0.0016=0.7
1.4382	59	0.0005=0.2	0.0017=0.75
1.4405	60	0.0006=0.25	0.0018=0.8
1.4428	61	0.0007=0.3	0.0019=0.85
1.4451	62	0.0008=0.35	0.0020=0.9
1.4474	63	0.0009=0.4	0.0021=0.9
1.4497	64	0.0010=0.45	0.0022=0.95
1.4520	65	0.0011=0.5	0.0023=1.0
1.4543	66	0.0012=0.5	0.0024=1.0
1.4567	67	...	...
1.4591	68	...	...
1.4615	69	...	...
1.4639	70	...	...
1.4663	71	...	...
1.4687	72	...	...
1.4711	73	0.0001=0.0	0.0015=0.55
1.4736	74	0.0002=0.05	0.0016=0.6
1.4761	75	0.0003=0.1	0.0017=0.65
1.4786	76	0.0004=0.15	0.0018=0.65
1.4811	77	0.0005=0.2	0.0019=0.7
1.4836	78	0.0006=0.2	0.0020=0.75
1.4862	79	0.0007=0.25	0.0021=0.8
1.4888	80	0.0008=0.3	0.0022=0.8
1.4914	81	0.0009=0.35	0.0023=0.85
1.4940	82	0.0010=0.35	0.0024=0.9
1.4966	83	0.0011=0.4	0.0025=0.9
1.4992	84	0.0012=0.45	0.0026=0.95
1.5019	85	0.0013=0.5	0.0027=1.0
1.5046	86	0.0014=0.5	0.0028=1.0
1.5073	87	...	...
1.5100	88	...	...
1.5127	89	...	...
1.5155	90	...	...

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[a] Intern. Sugar J., *10*: 69–70.

[b] Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

Table of corrections for the temperature.

Temperature of the prisms in ° C.	Dry substance.												
	0	5	10	15	20	25	30	40	50	60	70	80	90
	Subtract—												
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50
22	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
23	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
24	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
25	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
26	.12	.12	.13	.14	.14	.14	.15	.15	.16	.16	.16	.15	.14
27	.07	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.08	.07
	Add—												
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30	.12	.12	.13	.14	.14	.14	.15	.15	.16	.16	.16	.15	.14
31	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
32	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
33	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
34	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
35	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50

FOODS AND FEEDING STUFFS.

It is recommended—

That the referee for 1908–9 take up the question of acidity in cattle feeds and consider how the results obtained by current methods can be applied to agricultural problems.

Adopted.

DAIRY PRODUCTS.

It is recommended—

(1) That the following methods for the analysis of condensed milk be adopted as official:

(*1*) *Preparation of sample.* Place the can, if cold, in water at from 30° to 35° C. until warm. Open and mix thoroughly by transferring the contents of the can to some dish sufficiently large to thoroughly stir and make the whole mass homogeneous. Care must be taken to scrape out all milk adhering to the interior of the can. Weigh 100 grams into a 500 cc flask and make up to the mark with water. If the milk will not completely dissolve, each portion must be weighed out separately for analysis.

(*2*) *Total solids.* Use 10 cc of the above 20 per cent solution and proceed as in the case of milk analysis according to the method given in Bulletin 107, page 117, Method 1, drying either on sand or asbestos fiber.

(*3*) *Ash.* Ignite the residue from 10 cc of the 20 per cent solution at low red heat, leach with hot water if sucrose is present, ignite the residue and filter until white, add the leachings, evaporate to dryness again in usual manner and weigh.

(*4*) *Protein.* Determine nitrogen by Kjeldahl or Gunning method in 10 cc of the 20 per cent solution and multiply by 6.38.

(*5*) *Lactose.* Dilute 100 cc of the 20 per cent solution in a 250 cc flask to about 200 cc, add 6 cc of the Fehling's copper sulphate solution and make up to the mark, filter through a dry filter and determine lactose by Walker method, boiling only two minutes with the Fehling solution.

These methods were referred to the referee for 1908–9 for final recommendation and action by the association as to their adoption as official.

(2) That the methods for the determination of sucrose by inversion with citric acid and by inversion with hydrochloric acid be investigated by the referee for the ensuing year.

Adopted.

(3) That the determination of fat be studied, special attention being given to solutions of less than 20 per cent concentration.

Adopted.

(4) That the new Babcock standard, proposed by E. B. Holland and referred to Committee B, be referred to the referee for 1908–9.

Adopted.

This contemplated standard is as follows:

NEW BABCOCK STANDARD.[a]

SEC. 1. The unit of graduation for all Babcock glassware shall be the true cubic centimeter (0.998877 gram of water at 40° C.).

(*a*) With bottles, the capacity of each per cent on the scale shall be two-tenths (0.20) cubic centimeter.

(*b*) With pipettes and acid measures, the delivery shall be the intent of the graduation and the graduation shall be read with the bottom of the meniscus in line with the mark.

SEC. 2. The official method for testing Babcock bottles shall be calibration with mercury (13.5471 grams of clean, dry mercury at 20° C., carefully weighed on analytical balances, to be equal to 5 per cent on the scale), the bottle being previously filled to zero with mercury.

SEC. 3. Optional methods. The mercury and cork, alcohol and burette, and alcohol and brass plunger methods may be employed for the rapid testing of Babcock bottles, but the accuracy of all questionable bottles shall be determined by the official method.

SEC. 4. The official method for testing pipettes and acid measures shall be calibration by measuring in a burette the quantity of water (at 20° C.) delivered.

SEC. 5. The limit of error.

(*a*) For Babcock bottles, it shall be the smallest graduation on the scale, but in no case shall it exceed five-tenths (0.5) per cent, or for skim-milk bottles one-hundredth (0.01) per cent.

(*b*) For full quantity pipettes, it shall not exceed one-tenth (0.1) cubic centimeter, and for fractional pipettes, five hundredths (0.05) cubic centimeter.

(*c*) For acid measures, it shall not exceed two-tenths (0.2) cubic centimeter.

COMMITTEE C.—L. M. TOLMAN, CHAIRMAN.

(Food adulteration.)

WINES.

It is recommended—

(1) That a committee of five be appointed to cooperate with the Bureau of Standards in drawing up a standard alcohol table.

Adopted.

(2) That the question of a standard temperature of 20° for specific gravity and alcohol determinations be also referred to the committee of five.

Adopted.

(3) That the following subjects be given further study:

(a) Methods for determining glycerol.
(b) Methods for determining total fixed and volatile acids.
(c) Methods for determining coloring matter in genuine wines.

Adopted.

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[a] See also Twentieth Annual Report of the Massachusetts Agricultural Experiment Station, January, 1908, p. 113, for discussion of this standard.

FLAVORING EXTRACTS.

It is recommended—

That the colorimetric method for the determination of citral in lemon extract be adopted as provisional.

Adopted.

The method reads as follows:

DETERMINATION OF CITRAL IN LEMON EXTRACT.

Reagents.

Aldehyde-free alcohol.—Allow alcohol (95 per cent by volume) containing 5 grams of metaphenylene diamin hydrochlorid per liter to stand for twenty-four hours with frequent shaking. Nothing is gained by previous treatment with potassium hydroxid. Boil under a reflux cooler for at least eight hours, longer if necessary, allow to stand over night and distil, rejecting the first 10 and the last 5 per cent which come over. Store in a dark, cool place in well-filled bottles. Twenty-five cubic centimeters of this alcohol, on standing for twenty minutes in the cooling bath with the fuchsin solution (20 cc), should develop only a faint pink coloration. If a stronger color is developed, treat it again with metaphenylene diamin hydrochlorid.

Fuchsin solution.—Dissolve one-half gram of fuchsin in 250 cc of water, add an aqueous solution of sulphur dioxid containing 16 grams of the gas and allow to stand until colorless, make up to 1 liter with distilled water. This solution should stand twelve hours before using and should be discarded after three days.

Standard citral solution.—Use 1 mg of c. p. citral per cubic centimeter in 50 per cent by volume aldehyde-free alcohol. This solution deteriorates on standing and should not be kept over three or four days.

Apparatus.

A cooling bath.—Keep at from 14° C. to 16° C. The aldehyde-free alcohol, fuchsin solution, and comparison tubes are to be kept in this bath.

Colorimeter.—Any form of colorimeter using a large volume of solution and adapted to rapid manipulation may be used.

The comparison may also be made in Nessler or Hehner tubes.

Manipulation.[a]

Weigh in a stoppered weighing flask approximately 25 grams of extract, transfer to a 50 cc flask, and make up to the mark at room temperature with aldehyde-free alcohol. Measure at room temperature and transfer to a comparison tube 2 cc of this solution. Add 25 cc of the aldehyde-free alcohol (previously cooled in the bath), then 20 cc of the fuchsin solution (also cooled), and finally make up to the 50 cc mark with more aldehyde-free alcohol. Mix thoroughly, stopper, and place in the cooling bath for fifteen minutes. Prepare a standard for comparison at the same time and in the same manner, using 2 cc of the standard citral solution. Remove and compare the colors developed. Calculate the amount of citral present and repeat the determination, using a quantity sufficient to give the sample approximately the strength of the standard. From this result calculate the amount of citral in the sample. If the comparisons are made in Nessler tubes, standards containing 1, 1.5, 2, 2.5, 3, 3.5, and 4 mg should be prepared and the trial comparison made against these, the final comparison being made with standards between 1.5 and 2.5 mg varying but 0.25 mg.

DAIRY PRODUCTS.

It is recommended—

That the Baier and Neumann method for the detection of sucrate of lime in milk and cream be studied.

Adopted.

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[a] It is absolutely essential to keep the reagents and comparison tubes at the required temperature. Comparisons should be made within one minute after removing the tubes from the bath. Where the comparisons are made in the bath (this being possible only where the bath is glass), the standards should be discarded within twenty-five minutes after adding the fuchsin solution. Give samples and standards identical treatment.

The method reads as follows:

BAIER AND NEUMANN METHOD FOR SUCROSE IN MILK AND CREAM.

To 25 cc of milk or cream add 10 cc of a 5 per cent solution of uranium acetate, shake, allow to stand for five minutes and filter. If the filtrate is not clear pour back upon the same filter. To 10 cc of the clear filtrate (in the case of cream use the total filtrate if less than 10 cc) add 2 cc of a cold saturated solution of ammonium molybdate and 8 cc of diluted hydrochloric acid (1 part of 25 per cent hydrochloric acid to 7 parts of water), shake well, and place in a water bath at 80° C. for ten minutes. If the sample is pure the solution will resemble a nickel-sulphate solution, but if sugar is present it will be of a prussian-blue color. These colors usually appear after five minutes, and after ten minutes they are very intense.

DISTILLED LIQUORS.

It is recommended—

(1) That the modified Allen-Marquardt method for the determination of fusel oil be made a provisional method.

Adopted.

This modification, based on the determination of the amount of bichromate reduced in the oxidation of the higher alcohols, reads as follows:

MODIFIED ALLEN-MARQUARDT METHOD FOR FUSEL OIL.

Reagents and solution.

Thiosulphate solutions.—Make up two solutions of sodium thiosulphate, one an approximately three-fourths-normal strength not standardized, and the other a tenth-normal standardized against a standard potassium bichromate solution.

Carbon tetrachlorid.—Purify as directed in Bulletin 107, Rev., p. 98.

Potassium iodid solution.—Dissolve 1 gram for each cubic centimeter of water used.

Bichromate solution.—Dissolve 200 grams of pulverized potassium bichromate in 1,800 cc of water and add 200 cc of concentrated sulphuric acid.

Determination.

Proceed with the Allen-Marquardt method as given in Bulletin 107, Rev., page 98, to the point where the carbon tetrachlorid solution of the higher alcohols is ready to be oxidized, except that two washings with sodium sulphate are made. Add 50 cc of the oxidizing agent very carefully measured with pipette or burette and start the eight-hour oxidation. Take great care to prevent any isolation of spots of bichromate on the flask during the oxidation. Decomposition of the bichromate from overheating can best be prevented by slow boiling over several thicknesses of asbestos board. After the oxidation is complete, separate the bichromate solution from the carbon tetrachlorid in a separatory funnel, care being taken to wash the carbon tetrachlorid free from bichromate. Make up the bichromate solution to 500 cc.

Titration of bichromate solution.

Place 200 cc of this solution in a liter flask, add 20 cc of concentrated hydrochloric acid, 100 cc of potassium iodid solution, and 50 cc of the approximately three-fourths-normal thiosulphate. Make this last addition by means of a burette. (If a high content of fusel oil is present, 50 cc of thiosulphate may be excessive and a smaller amount should be used, the same quantity being added to the sample and to the blank.) Run blanks containing exactly the same amount of reagents with each series, and treat them in the same way, starting them at the point where the carbon tetrachlorid is washed with sodium chlorid. The titration of this blank, to which has been added exactly the same amount of three-fourths-normal thiosulphate, gives the value of the bichromate solution. The difference in cubic centimeters of tenth-normal thiosulphate used in titrating the blank and the samples gives the amount of bichromate reduced by the higher alcohols. This difference in cubic centimeters of tenth-normal thiosulphate multiplied by the factor 0.001773 gives grams of higher alcohols present.

(2) That in the present method (Bul. 107, Rev., p. 98) a second washing with sodium sulphate be prescribed.

Adopted.

(3) That the following method for the determination of water-insoluble color be made provisional.

Adopted.

DETERMINATION OF WATER-INSOLUBLE COLOR IN WHISKIES—PROVISIONAL.

Evaporate 50 cc of the sample just to dryness. Take up with cold water, using approximately 15 cc, filter, and wash until the filtrate amounts to nearly 25 cc. To this filtrate add 25 cc of absolute alcohol or 26.3 cc of 95 per cent by volume alcohol, and made up to 50 cc by the addition of water. Mix thoroughly and compare in a colorimeter with the original material. Calculate the per cent of color insoluble in water from these readings.

(4) That the modified Marsh test for the quantitative determination of the color insoluble in amyl alcohol be adopted as a provisional method.

Adopted.

MODIFIED MARSH TEST FOR THE QUANTITATIVE DETERMINATION OF COLOR INSOLUBLE IN AMYL ALCOHOL—PROVISIONAL.

Evaporate 50 cc of the whisky just to dryness on the steam bath. Add 26.3 cc of 95 per cent alcohol to dissolve the residue. Transfer to a 50 cc flask and make up to volume with water to obtain a uniform alcohol concentration. Place 25 cc of this solution in a separatory funnel and add 20 cc of the Marsh reagent, shaking lightly so as not to form an emulsion. (This reagent consists of 100 cc of pure amyl alcohol, 3 cc of sirupy phosphoric acid, and 3 cc of water; shake before using.) Allow the layers to separate and repeat this shaking and standing twice again. After the layers have clearly separated draw off the lower or watery layer which contains the caramel into a 25 cc cylinder and make up to volume with 50 per cent by volume alcohol. Compare this solution in a colorimeter with the untreated 25 cc. Calculate the result of this reading to the per cent of color insoluble in amyl alcohol.

(5) That the provisional Roese method for determining fusel oil (Bul. 107, Rev., p. 97) be dropped.

Adopted.

Spices.

It is recommended—

That methods for the detection of added oil in paprika be further studied.

Adopted.

Meat and Fish.

It is recommended—

(1) That the study be continued in an attempt to apply, improve, or devise methods for the most accurate separation possible of the various protein bodies in meat.

(2) That the method for determining ammoniacal nitrogen by distillation under reduced pressure be compared with the magnesium oxid method now generally used. (Bul. 107, p. 9.)

Cereal Products.

It is recommended—

That methods applicable for the separation of the gluten constituents of flour be studied, tests to be made upon the several grades, as patents, first and second clears, and on flours produced from different varieties and types of wheat.

Adopted.

CANNED VEGETABLES.

It is recommended—

That methods for the detection of soaked peas be further studied.

Adopted.

MEAT PROTEIDS.

It is recommended—

That the work on the separation of meat proteids be continued along the lines pursued during the past year.

Adopted.

[The work included analyses of cold-water extracts prepared under known conditions from different wholesale cuts; the relation of acidity to the ripening of meat is also mentioned.]

TEA, COFFEE, AND COCOA.

It is recommended—

(1) That methods for the estimation of caffein be further studied. (Original Gomberg method, J. Amer. Chem. Soc., 1896, p. 331, and modifications.)

Adopted.

(2) That the Dubois method for the determination of sugars in chocolate be further studied (J. Amer. Chem. Soc., 1907, *29:* 556; Bul. 107, Rev. p. 256.)

(3) That the Doolittle and Woodruff method (Bul. 105, p. 48) for extract in tea be substituted for the Krauch method (Bul. 107, p. 149, sec. 5) as provisional.

Adopted.

COLORS.

It is recommended—

(1) That an effort be made to obtain authentic samples of vegetable or natural coloring matters such as are used in food products.

(2) That a study be made of the characteristics of vegetable coloring matters and methods of identification.

(3) That pure colors be synthetically prepared to serve as standards.

(4) That the separation and identification of mixed colors be studied.

These recommendations were adopted.

SPECIAL ACTION TAKEN.

The changes in the constitution proposed in 1907 (see Circular 38, page 10, or Bulletin 116, page 108) were adopted. These changes in effect extend formal membership to Canadian and Mexican official chemists, making the association a North American assembly rather than one confined to the United States.

It will be noted by reference to the list of referees that three new positions were created as follows—

An associate referee to consider the definition of the term "available potash."

Two associate referees on drugs.

A referee and an associate referee on waters, to study methods for the analysis of mineral, sanitary, irrigation, and technical waters.

The following resolution indorsing official legislation regulating the composition and sale of insecticides and fungicides was adopted:

> Whereas, a National bill to regulate the composition and sale of insecticides and fungicides has been recently drawn up by a committee composed of members of the Association of Economic Manufacturers of Insecticides and agricultural chemists interested in insecticide and fungicide analysis, which bill will be presented to Congress for approval and passage, the Association of Official Agricultural Chemists does hereby express its approval of National legislation on this subject, which legislation it is believed will be of inestimable

service in protecting the farming community as well as the legitimate manufacturer and in unifying State insecticide and fungicide laws.

Two new committees were created, as follows: (1) A committee of five on standardization of alcohol tables; (2) a committee of three to cooperate with similar committees from other organizations in this country in unifying methods for the analysis of fats and oils, with a view to the establishment of an international commission for this purpose.

The resolution creating a standing committee on recommendations of referees and revision of methods adopted in 1907 went into effect at the 1908 meeting, and the membership of the committee is given in the appended list.

Attention is called to the following provisions of the resolution affecting the forwarding of recommendations to the chairmen of the several subcommittees in advance of the meeting:

> *Resolved further*, That this committee shall organize from its membership three subcommittees, corresponding to the present committees A, B, and C, to whom all recommendations of new methods or of changes in methods, provisional or official, shall be submitted not later than three weeks prior to the date of the meeting of the association at which it is desired that the recommendations in question shall be considered, and said committee on recommendations shall report to the association, with its approval, disapproval, or suggestion of amendment, all recommendations submitted in due form for its consideration.

The question of the adoption and printing of conversion tables was referred to this committee.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1908–9.

President.

W. D. Bigelow, Washington, D. C.

Vice-President.

W. A. Withers, Raleigh, N. C.

Secretary.

H. W. Wiley, Washington, D. C.

Additional members of the Executive Committee.

E. F. Ladd, Agricultural College, N. Dak.
E. B. Holland, Amherst, Mass.

Referees.

Phosphoric acid: W. F. Hand, Agricultural College, Miss.
Nitrogen:
  Determination of nitrogen: C. H. Jones, Burlington, Vt.
  Separation of nitrogenous bodies: P. F. Trowbridge, Columbia, Mo. (meat proteids).
Potash: B. B. Ross, Auburn, Ala.
Soils: S. D. Averitt, Lexington, Ky.
Dairy products: J. M. Bartlett, Orono, Me.
Foods and feeding stuffs: J. P. Street, New Haven, Conn.
Food adulteration: H. E. Barnard, Indianapolis, Ind.
Sugar: A. H. Bryan, Washington, D. C.
Tannin: F. P. Veitch, Washington, D. C.
Insecticides: C. C. McDonnell, Washington, D. C.
Inorganic plant constituents: Floyd W. Robison, Lansing, Mich.
Medicinal plants and drugs: L. F. Kebler, Washington, D. C.
Water: J. K. Haywood, Washington, D. C.

Associate referees.

Phosphoric acid: H. D. Haskins, Amherst, Mass.
Nitrogen:
  Determination of nitrogen: J. W. Kellogg, Harrisburg, Pa.
  Separation of nitrogenous bodies—
    Milk and cheese: G. E. Patrick, Washington, D. C.
    Vegetable proteids: R. Harcourt, Guelph, Canada.
Potash:
  E. L. Baker, Geneva, N. Y.
  Jas. A. Bizzell, Ithaca, N. Y. (special associate referee on "available potash").
Soils: J. G. Lipman, New Brunswick, N. J.
Dairy products: L. G. Michael, Ames, Iowa.
Foods and feeding stuffs: F. W. Morse, Durham, N. H.
Food adulteration:
  Colors: H. M. Loomis, Seattle, Wash.
  Saccharine products: Chas. D. Howard, Concord, N. H.
  Fruit products: C. B. Cochran, Westchester, Pa.
  Wine: Julius Hortvet, St. Paul, Minn.
  Beer: H. E. Barnard, Indianapolis, Ind.
  Distilled liquors: L. M. Tolman, Washington, D. C.
  Vinegar: R. W. Balcom, New York, N. Y.
  Flavoring extracts: E. M. Chace, Washington, D. C.
  Spices: A. F. Seeker, New York, N. Y.
  Baking powders and baking chemicals: Edmund C. Clark, Boston, Mass.
  Meat and fish: F. C. Weber, Washington, D. C.
  Fats and oils: T. J. Bryan, Chicago, Ill.
  Dairy products: Hermann C. Lythgoe, Boston, Mass.
  Cereal products: E. F. Ladd, Agricultural College, N. Dak.
  Vegetables: W. L. Dubois, Buffalo, N. Y.
  Condiments other than spices: H. E. Bishop, Indianapolis, Ind.
  Cocoa and cocoa products: A. G. Woodman, Boston, Mass.
  Tea and coffee: A. G. Woodman, Boston, Mass.
  Preservatives: P. B. Dunbar, Washington, D. C.
  Determination of water in foods: H. C. Whittier, Wooster, Ohio.
Sugar:
  Molasses methods: H. P. Agee, New Orleans, La.
  Chemical methods: A. H. Bryan, Washington, D. C.
Tannin: C. F. Sammet, Washington, D. C.
Insecticides: Howard R. Watkins, Washington, D. C.
Inorganic plant constituents: O. M. Shedd, Lexington, Ky.
Medicinal plants:
  C. H. La Wall, Philadelphia, Pa.
  H. H. Rusby, New York, N. Y.
Water: W. W. Skinner, Washington, D. C.

SPECIAL COMMITTEES.

Food Standards.

Mr. William Frear, State College, Pa., chairman.
Mr. H. W. Wiley, Washington, D. C.
Mr. H. A. Weber, Columbus, Ohio.
Mr. M. A. Scovell, Lexington, Ky.
Mr. E. H. Jenkins, New Haven, Conn.

Fertilizer Legislation.

Mr. H. W. Wiley, Washington, D. C., chairman.
Mr. B. W. Kilgore, Raleigh, N. C.
Mr. H. B. McDonnell, College Park, Md.
Mr. J. L. Hills, Burlington, Vt.
Mr. B. B. Ross, Auburn, Ala.

Testing Chemical Reagents.

Mr. L. F. Kebler, Washington, D. C., chairman.
Mr. A. L. Winton, Chicago, Ill.
Mr. B. W. Kilgore, Raleigh, N. C.

Committee to present the question of the unification of terms to the International Congress of Applied Chemistry.

Mr. R. J. Davidson, Blacksburg, Va., chairman.
Mr. C. G. Hopkins, Urbana, Ill.
Mr. W. D. Bigelow, Washington, D. C.
Mr. G. S. Fraps, College Station, Tex.
Mr. B. W. Kilgore, Raleigh, N. C.
Mr. H. J. Wheeler, Kingston, R. I.
Mr. J. T. Willard, Manhattan, Kans.

Committee on Standardization of Alcohol Tables.

Mr. L. M. Tolman, Washington, D. C., chairman.
Mr. M. E. Jaffa, Berkeley, Cal.
Mr. A. B. Adams, Washington, D. C.
Mr. R. J. Davidson, Blacksburg, Va.
Mr. H. E. Barnard, Indianapolis, Ind.

Committee on Unification of Methods of Analysis of Fats and Oils.

Mr. L. M. Tolman, Washington, D. C., chairman.
Mr. P. H. Walker, Washington, D. C.
Mr. A. Lowenstein, Chicago, Ill.

Standing Committee on Recommendations of Referees and Revision of Methods.

(The figures in parentheses refer to number of years appointee is to serve.)

COMMITTEE A: B. B. Ross (3), J. P. Street (2), *J. K. Haywood (1), chairman, Bureau of Chemistry, Washington, D. C.*

COMMITTEE B: E. M. Chace (3), *F. W. Woll (2), chairman, Agricultural Experiment Station, Madison, Wis.;* F. P. Veitch (1).

COMMITTEE C: C. D. Howard (3), *A. L. Winton (2), chairman, U. S. Food Inspection Laboratory, Chicago, Ill.;* L. M. Tolman (1).

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